

practical NO. 1

01

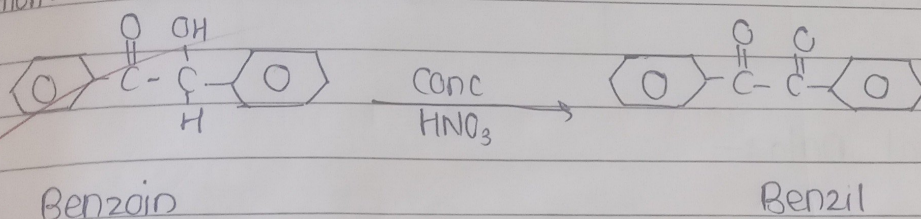
Benzoin \rightarrow Benzil \rightarrow Benzilic acid.

part-I

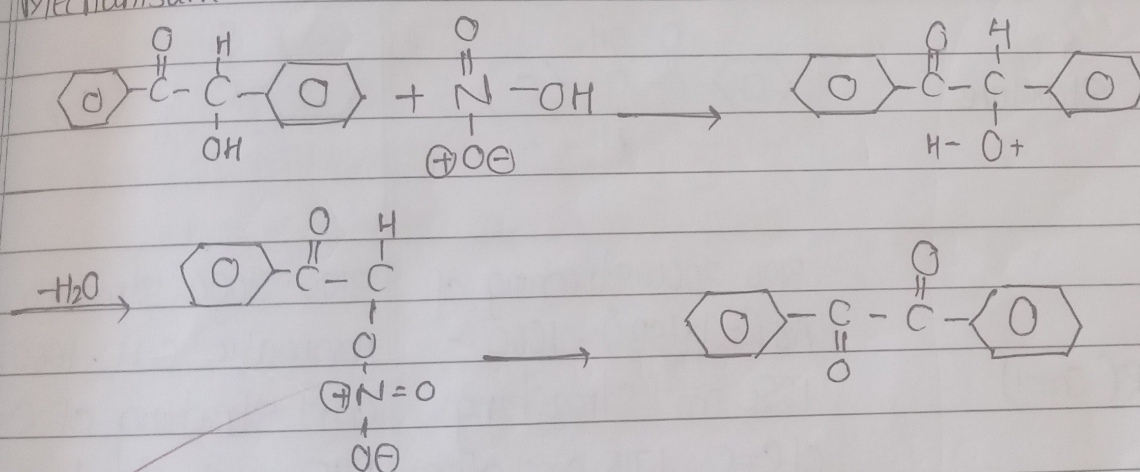
Aim:- preparation of Benzil from Benzoin.

Requirement :- Conical flask, burner, Condensers, Benzoin, Conc HNO_3 etc.

Reaction:



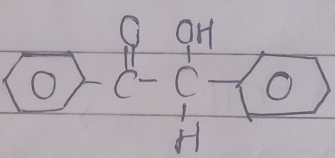
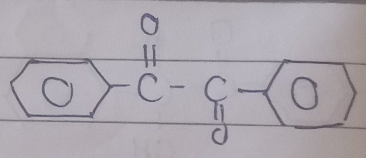
Mechanism



Stoichiometric Calculation.

	Benzoin	Con. HNO_3
Molecular formula	$\text{C}_{14}\text{H}_{12}\text{O}_2$	$\text{H}_1\text{N}_1\text{O}_3$
Molar weight	212	63
Molar ratio	1	1
Volume	-	0.75
No. of moles	0.018	0.018
ϕ (gm)	4 gm	1.34 gm

Spectral Data :-

	Benzoin	Benzil
Structure		
IR (cm^{-1})	3100-3000 stretching of Aromatic C-H (sp^2) - 1660 - 1550 cm^{-1} stretching of C=C. 1715 cm^{-1} stretching of Ketonic C=O	3100-3000 stretching of Aromatic C-H. 1660-1550 cm^{-1} stretching of C=C. 1715 cm^{-1} stretching of Ketonic C=O.
$^1\text{H NMR}$	Benzene ring 6.5-8.5 δH R-OH : 1-6.	Benzene ring 6.5-8.5 m 8H.

procedures:-

Take 4 gm of benzoic acid and 20 cm³ of con HNO₃ in Conical flask.
 Heat it on boiling water bath with occasional stirring until the evolution of oxides of nitrogen has stopped.
 pour the reaction mixture in 20 cm³ of cold water and stir well.
 The oil crystallises and yellow solid is obtained.
 Filter the crude product at the suction pump wash with cold water to remove HNO₃ and dry. Note the yield of the crude benzoic acid.
 Purify the product and take its mp.

Calculation:

Weight of crude product: 3.7 gm
 Melting point : 94°C

Theoretical yield:-

212 gm of benzoic acid = 210 gm of Benzoic acid

4 gm of benzoic acid = x gm of Benzoic acid

$$x = \frac{210 \times 4}{212}$$

$$x = 3.96 \text{ gm}$$

Percentage yield: $\frac{\text{practical yield}}{\text{Theoretical yield}} \times 100$

$$= \frac{3.7}{3.96} \times 100$$

$$= 96\%$$

$$\text{RF value} = \frac{\text{Distance travelled by solute}}{\text{Distance travelled by solvent}}$$

$$= \frac{3.5}{4}$$

$$= 0.87$$

MSDS Data:-

Name of compound	Health Hazards	First Aid.
Benzoin	many causes eyes and skin irritation. Causes respiratory tract irritation Harmful if Swallowed.	Flush the eyes and skin with plenty of water.
Nitric acid	Causes severe eyes and skin burns. may causes respiratory tract and digestive tract irritation Very harmful if Swallowed.	Rinse the eyes with plenty of water flush the affected area of skin with plenty of water.
Ethyl alcohol	Cause eye skin respiratory tract irritation.	Flush eyes and skin with plenty of water.

Result:-

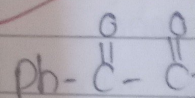
Theoretical
Melting point
Weight of
percentage

Aim:- prep

Requirement

Reaction.

Mechanism



→

Result:-

03

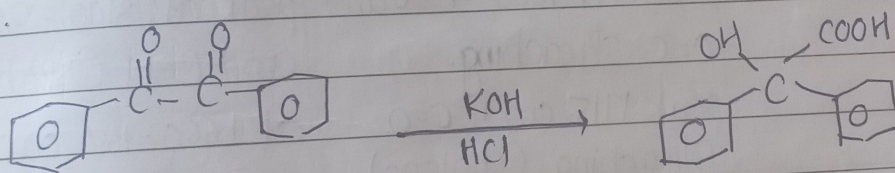
Theoretical yield: 3.96 gm
Melting point : 94
Weight of the product: 3.7
percentage yield: 96%

Part - II

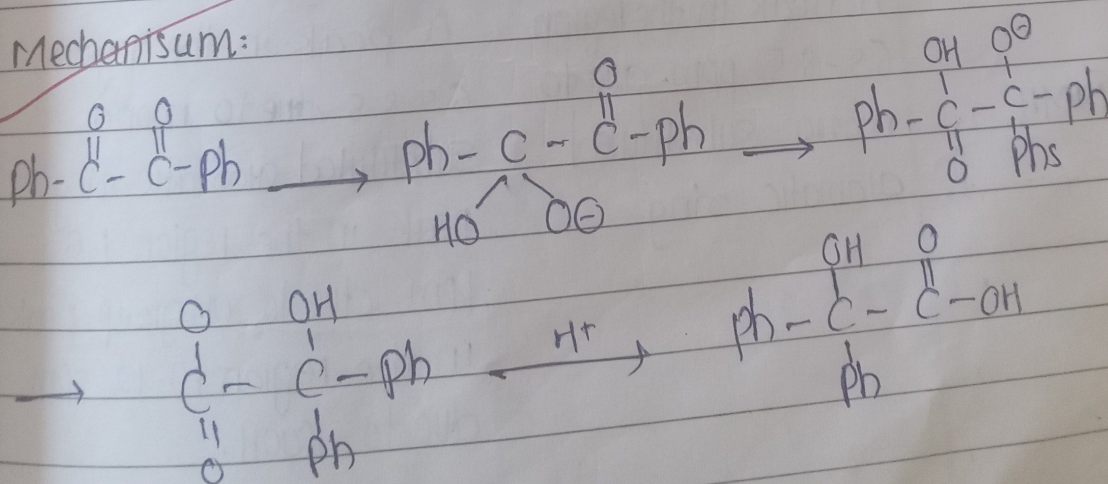
Aim:- preparation of Benzilic acid from benzile.

Requirement: Conical flask, 1g Benzile, 150 cm³ Water bath cold
Water Filter paper.

Reaction:



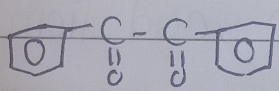
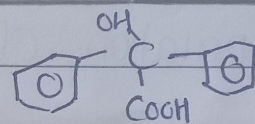
Mechanism:



Stoichiometric Calculation:

	Benzil	KOH
Molecular formula.	$C_{14}H_{10}O_2$	$K_2O.H_2$
Molecular weight	210	56
Molar ratio	1	1
ϕ (gm)	1	0.280
Volume	-	-
N.O. of moles.	0.005	0.005

Spectral Data:-

Structure		
IR (cm^{-1})	<p>peak at 3100-3000 cm^{-1} C-H (sp^2) stretching</p> <p>peak at 1600-1550 cm^{-1} C=C stretching.</p> <p>peak 1715 cm^{-1} C=O stretching (Ketone)</p>	<p>peak at 3600-3200 cm^{-1} O-H stretching</p> <p>peak at 3100-3000 cm^{-1} C-H (sp^2) peak</p> <p>at 1660-1550 cm^{-1} C=C.</p>
1H NMR	<p>A peak in the region 6.5-8.5 ppm. due to mono substituted aromatic ring</p>	<p>A peak in the region 6.5-8.5 due to mono substituted aromatic ring</p> <p>peak in the region 1-6 ppm due to OH group peak</p> <p>in the region 10-12 ppm due to COOH.</p>

procedure:-

Reflux the mixture 1 gm of benzil 1 g of KOH 5 cm³ of water and 2.5 cm³ of alcohol on heat for 10-15 min

pour the content of the flask into a pore dish allow it to cool The potassium salt of benzoic acid and crystallized.

Filter off the crystal at the pump and wash with ice cold alcohol.

Dissolved the potassium salt in about 10 cm³ of water and acidify with con. HCl to get precipitate wash the precipitate and recrystallize with hot water take the melting point of product.

check the purity by TLC. Submit the product.

Benzilic acid not red dye white colour
Calculation.

Theoretical yield:-

210 g of benzil = 228 g of benzoic acid.

1 g of benzil = x g of benzoic acid

$$x = \frac{228 \times 1}{210}$$

x = 1.08 Benzoic acid.

Percentage yield:-

$$\frac{\text{practical yield}}{\text{Theoretical yield}} \times 100$$

$$= \frac{0.6}{1.08} \times 100$$

$$= 55\%$$

RF value:-

Distance travelled by solute
Distance travelled by solvent

$$= \frac{3.2}{5}$$

$$= 0.64$$

non-polar (n-hexane) = 80%
polar (ethyl acetate) = 20%

Result:

Theoretical yield = 1.08 gm

Percentage yield = 5%

Weight of the product = 0.69 gm

RF value = 0.64

Solvent System: n-hexane - 80%
ethyl acetate - 20%

MSDS Data:-

Name of Compound	Health Hazards	First Aid.
Benzil	eyes and skin irritation Causes respiratory tract irritation Harmful if swallowed	Flush the eyes and skin with water. Call poison center immediately
potassium Hydroxide.	Causes severe skin burns and eyes damage Causes respiratory and digestive tract irritation.	Flush the eyes with plenty of water and move to air at least 15 min
HCl	Causes severe skin burns and eyes damage Causes respiratory tract irritation.	Flush the eye plenty of water and move to at least 15 min.

[Signature]
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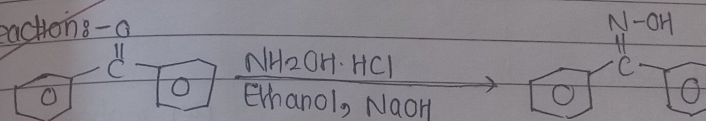
Benzophenone \rightarrow Benzophenone oxime \rightarrow Benzanilide.

part-I

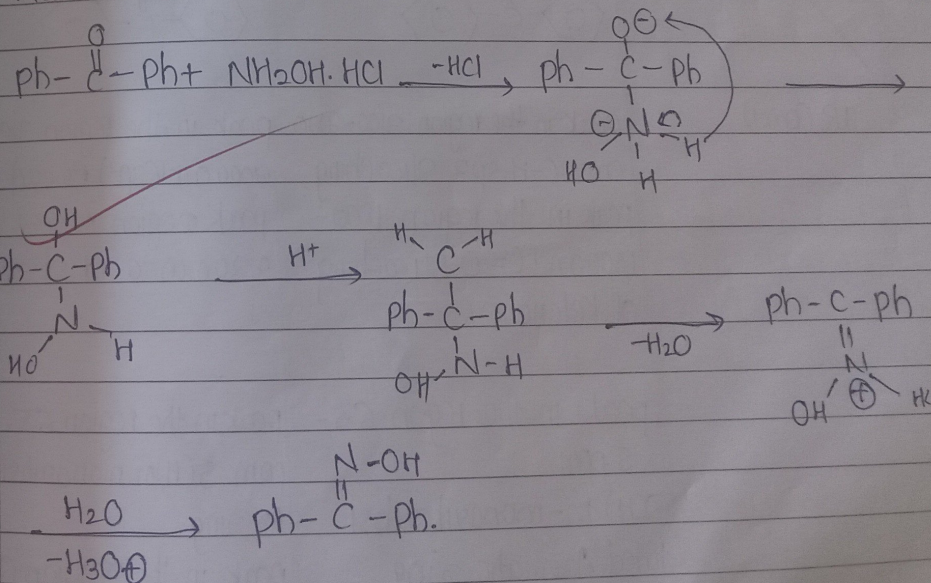
Aim:- preparation of Benzophenone oxime from Benzophenone.

Requirment:- RBF, burnner, water bath, benzophenone, xestified spirit, hydroxylamine, hydrochloride.

Reaction:-



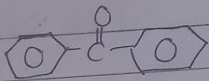
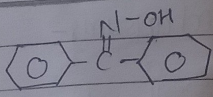
Mechanism:-



Stoichiometric Calculation.

	Benzophenone	Hydroxylamine Hydrochloride
Molecular formula	$C_{13}H_{10}O$	NH_2OCl
Molecular weight	182	69.5
Molar Ratio	1	1
Quantity (gm)	3	1.112
Quantity (cm ³)	-	0.6825
no. of moles	0.016	0.016

Spectral Data:

	Benzophenone	Benzophenone Oxime
Structure		
IR (cm ⁻¹)	<p>peak in the region 3100-3000 cm⁻¹ C-H sp² stretching</p> <p>peak in the region 1800-1600 cm⁻¹ C=O stretching of ketone.</p>	<p>peak in the region 3100-3000 cm⁻¹ (sp²) Broad</p> <p>peak region 3600-3200 cm⁻¹ O-H stretching</p>
	<p>peak in the region 6.5-8.5 ppm</p> <p>5H(m)-monosubstituted Aromatic ring</p> <p>peak in the region 6.5-8.5 ppm 5H(m) monosubstituted Aromatic ring</p>	<p>peak in the region 6.5-8.5 ppm 5H(m) monosubstituted Aromatic ring</p> <p>peak in the region 6.5-8.5 ppm 5H(m) monosubstituted Aromatic ring</p>

procedure:-

Take 3g of Benzophenone 2 grams of hydroxylamine hydrochloride and 6cm³ of rectified spirit.

Add 1 cm³ of water to this mixture add 2.5 grams of solid NaOH in portion of 0.5 grams with constant shaking

the reflux the reaction mixture 10 mint. cool and pour the content of the flask into a mixture of pour the content of the flask

into a mixture of 10cm³ of conc. HCl and add 60cm³ of water. Filter the precipitated and dry the product.

Note the yield of crude benzophenone oxime purify the portion of the product and take its melting point.

observation:

Weight of crude product: 2.44gm

Melting point: 142°C

Calculation:-

Theoretical yield:-

182g of Benzophenone = 197g of Benzophenone oxime

3g of Benzophenone = x

$$x = \frac{197 \times 3}{182}$$

$$x = 3.2g$$

Percentage yield = $\frac{\text{Observed yield}}{\text{Theoretical yield}} \times 100$

$$= \frac{2.44}{3.2} \times 100 = 75\%$$

Preparation of benzil from benzoin
benzoin + 20 cm³ conc HNO₃ in conical
at bp boiling H₂O bath 5 hr

Result:-

Weight of crude product:- 2.44 gm
percentage yield :- 75%
Theoretical yield :- 3.2 g
Melting point :- 142°C.

MSDS Data:-

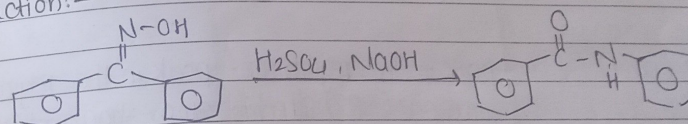
Name of Compound	Health Hazard's	First Aid.
Benzophenone.	Causes eyes and skin irritation causes gastrointestinal irritation. Causes respiratory tract irritation.	Flush eyes with plenty water for at least 15 mint move in fresh air
Ethanol	Causes eye and skin irritation causes respiratory	Flush eyes with plenty water move to fresh air
Hydroxyl amine hydroxy chloride	Causes eyes and skin irritation Harmful if Swallowed many Cause respiratory irritation	Flush eyes with water and move to fresh air at least 15 mins.

Part-II

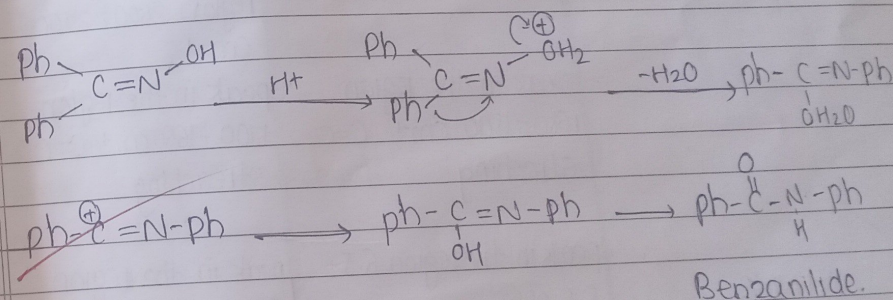
Aim:- preparation of Benzanilide from Benzophenone oxime

Requirements:- Beakers, Phenazophenone oxime, water bath
diethyl ether, penta Chloride

Reaction:-



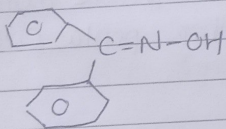
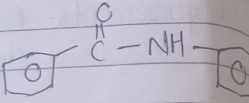
Mechanism:-



Stoichiometric Calculation:-

	Benzophenone oxime	Conc H ₂ SO ₄
Molecular formula	C ₁₃ H ₁₁ NO	H ₂ SO ₄
Molecular weight	197	98
Molar ratio	1	1
W (gm)	1	0.49
Volume	-	0.26
no. of moles.	0.005	0.005

Spectral Data:-

	Benzophenone oxime	Benzanilide
Structure:-		
IR (cm ⁻¹)	<p>peak in the region 3100-3000 C-H (sp²) stretching</p> <p>Broad peak in the region 3600-3200 cm⁻¹</p> <p>peak in the region 1680-1500 cm⁻¹ C=C stretching</p>	<p>peak in the region 3100-3000 cm⁻¹ C-H (sp²) stretching</p> <p>sharp peak in the region 3600-3200 cm⁻¹</p> <p>peak in the region 1700-1680 cm⁻¹ C=O stretching</p>
¹ H NMR	<p>peak in the region 6.5-8.5 ppm.</p> <p>peak in the region 1-6 ppm.</p> <p>H(s) OH proton.</p>	<p>peak in the region 6.5-8.5 ppm 5H(m) monosubstituted Ar-ring</p> <p>aromatic ring</p> <p>peak in the region ppm Ar-NH proton 1H(s)</p>

procedure:-

Take 1 gram of Benzophenone oxime add the cool solution of conc. H₂SO₄ in water (1.6 cm³ conc. H₂SO₄ to 2.0 cm³ water) shake the flask and warm it gently to begin the reaction. Remove the flask from burner and allowed reaction on to continue. Once the reaction subsides. Cool the content flask and then add 4 cm³ of water.

Add 10N NaOH solution to reaction mixture dropwise maintaining compound by using 3 portions of carbon tetrachloride. yellow oil is obtained which solidified on cooling purify the compound by recrystallisation.

Note the yield of Benzanilide. take the melting point of the the product.

Check the purify the product by TLC. Submit the dried product.

Observation:

Weight of the Benzanilide:- 0.2 gm

Melting point :- 162°C

Calculation:

Theoretical yield:-

197 g of Benzophenone = 197 g of Benzanilide oxime.

1 g of Benzophenone oxime : x g of Benzanilide

$$x = \frac{197 \times 1}{197}$$

x = 1 g of benzanilide.

Preparation of benzil from benzoin
 benzoin + 20 cm³ conc HNO₃ in conical
 flask on boiling H₂O bath until

DATE: 26/04/24

percentage yield = $\frac{\text{Weight of the product}}{\text{Theoretical yield}} \times 100$

$$= \frac{0.2}{1} \times 100$$

$$= 20\%$$

Rf value:

$\frac{\text{Distance traveled by solute}}{\text{Distance traveled by solvent}}$

$$= \frac{3.2}{4.0}$$

$$= 0.8$$

Result:-

Weight of the crude product:-	0.2 grams
Theoretical yield	:- 1g
percentage yield	:- 20%
Melting point	:- 162°C
Rf value	:- 0.8
Solvent System	:- 60% (non-polar) 40% (polar)

MSDS Data:

Name of compound	Health Hazard	First Aid.
Benzophenone oxime	may cause eyes irritation. may cause digestive tract irritation. Harmful if swallowed	Flush eyes with plenty of water get medical aid
Diethyl ether	may cause skin dryness. Harmful if swallowed	Flush eyes and skin with plenty of water get medical aid
phosphorous penta Chloride	Chemical burns of the respiratory tract Harmful if swallowed weird	get medical aid immediately if irritating is difficult give oxygen.

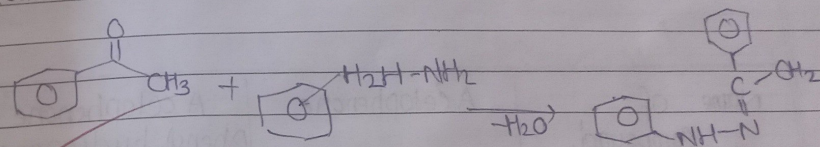
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part-I

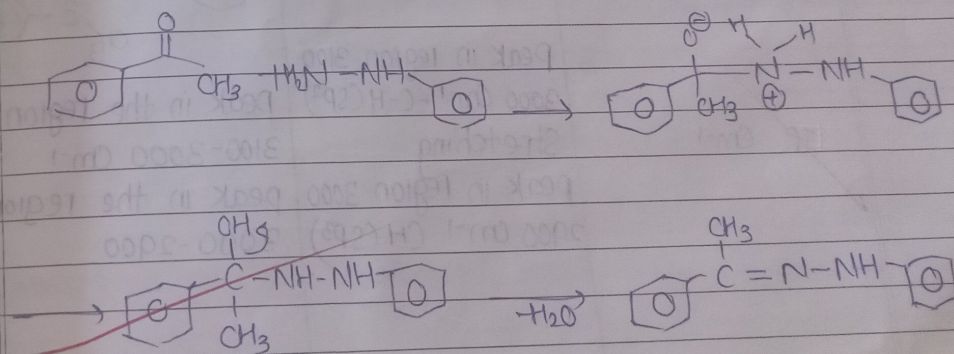
Aim:- Preparation of Acetophenone phenylhydrazone from Acetophenone.

Requirement: Beakers, Acetophenone phenylhydrazone, glacial acetic acid etc.

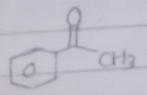
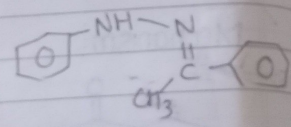
Reaction:



Mechanism:



	Acetophenone	phenylhydrazine
Molecular formula	C_8H_8O	$C_6H_5N_2$
Molecular weight	120	108
Molar ratio	1	1
Quantity in (g)	5.14	4.20
Volume	5	0.428
Moles	0.042	

Name of compound	Acetophenone	Acetophenone phenyl hydrazone
Structure		
IR cm^{-1}	peak in region 3100 $3000\text{ }cm^{-1}$ - C-H (sp^2) Stretching peak in region 3000 $2900\text{ }cm^{-1}$ C-H (sp^3)	peak in the region $3100-3000\text{ }cm^{-1}$ peak in the region $3000-2900$
$^1H\text{ NMR}$	peak in the region ppm 5-7 m-mono-substituted Aromatic peak in the region 2-3 ppm 3H (s) methyl	peak in the region 1-2 ppm 3H (s) alkyl proton.

procedures:-

Take 5 cm^3 acetophenone and 5 cm^3 phenyl hydrazine with 30 cm^3 of ethanol few drops of glacial acetic acid is formed

Warm the reaction mixture till the solid product is formed cool and filter and wash the product with dil HCl followed by about 10 cm^3 of cold distilled spirit Dry the product

Note the yield of crude acetophenone phenyl hydrazone purify small portion of the product and take its M.P

ethanol
recrystalline

Observation:-

Weight of the crude product: 6.9 gm

physical Constant : $186^\circ C$

Calculation:

120 gm of acetophenone - 210 gms of acetophenone phenyl hydrazone
 5.14 gm of acetophenone - x gm of acetophenone phenyl hydrazone

$$x = \frac{210 \times 5.14}{120}$$

$$x = 8.99 \text{ gms.}$$

percentage yield: $\frac{\text{Observed yield}}{\text{Theoretical yield}} \times 100$

$$= \frac{6.9}{8.99} \times 100$$

$$= 75\%$$

- heat on boiling H_2O bath $50^\circ C$ in conical
 - Pour in 200ml cold H_2O until evolution
 - oil phase

Result:-

Weight of Crude product: 6.9 gms
 Theor. yield : 8.99 gms
 Percentage yield : 76.75%
 Physical constant : 186°C.

MSDS Data:

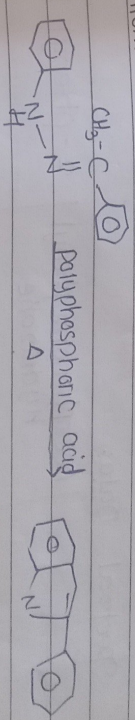
Name of compound	Health hazards	First Aid.
Acetophenone	Cause eyes and skin irritation Causes respiratory irritation	Flush the eye and skin with plenty of water move to fresh air and get medical Aid
phenyl hydrazine	Causes eyes and skin irritation Causes respiratory tract and digestive tract irritation	Flush the eyes skin with plenty of water for least 15 minit
Ethyl Alcohol	Causes eyes and skin irritation Causes respiratory tract irritation	Wash skin and eyes with plenty of water.
Acetic acid.	Causes severe skin burns and eye damage may be harmful	Wash affected area with plenty of water to fresh air.

Part - II

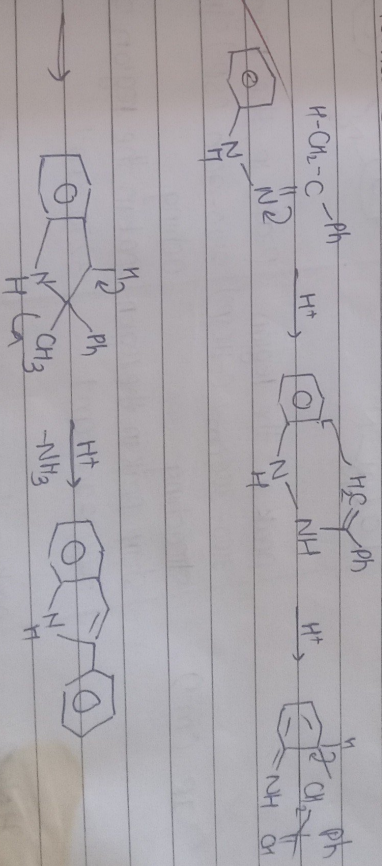
Aim: preparation of phenyl indole from acetophenone phenyl hydrazine.

Requirement: Acetophenone phenyl hydrazine, Beaker cold water, water bath.

Reaction:



Mechanism:

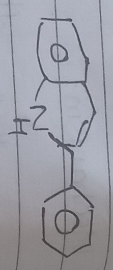
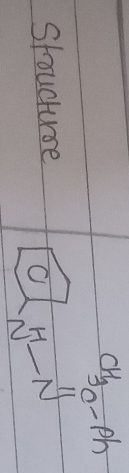


2-phenyl indole

Heat on boiling H₂O bath until evolution of H₂O is complete.

Stoichiometric calculation	
Calculation	H ₂ N + 2 PhNO ₂ →
Molecular formula	C ₁₄ H ₁₁ N ₃
Molecular weight	210
Molar Ratio	1
Wt (gm)	1.588
Volume	0.756
No. of moles	0.0047

Spectral Data: Acetophenone phenyl Hydrazone 2-phenyl Indole



IR (cm⁻¹)

peak in the region 3100-3000 cm ⁻¹ (C-H sp ²)	peak in the region 3100-3000 (sp ²) SH stretching
sharp peak in the region 3600-3200 cm ⁻¹	peak in the region 3200 cm ⁻¹

¹H NMR

peak in the region 6.5 to 8.5 ppm	peak in the region 6.5 to 8.5 (SH)
peak in the region 8-5 ppm	m (monosubstituted) peak 6.5 to 8.5 SH

Take 1 gm of crude acetophenone phenyl hydrazone and 3.5 cm³ of polysulphuric acid. Heat the mixture with constant stirring and maintain temperature 100-120 for 5 min. Filter the product and stir well to get product. Purify the small portion and take TLC.

Weight of the Product : 0.4 gm
Melting point : 190°C

210 gm of acetophenone phenyl hydrazone = 193 gm of 2-phenyl indole
1 gm of acetophenone phenyl hydrazone = 8.9 gm of 2-phenyl indole

$$x = \frac{193 \times 1}{210}$$

$$x = 0.919$$

$$= \frac{\text{Weight of the product}}{\text{theoretical yield}} \times 100$$

$$= \frac{0.4}{0.919} \times 100$$

$$= 43.58\%$$

heat on boiling H₂O bath in circular
- Pour in 200ml cold H₂O
- at 100°C

RF value: Distance travelled by solute
Distance travelled by solvent

$\frac{9.4}{4.8}$

0.74

Weight of the product: 0.4gm
Theoretical yield: 0.919 gms
Practical yield: 48.53%
Physical constant: 190°C
RF value: non-polar 80%
polar 20%

MSDS Data:

Compound	Health Hazards	First Aid
Acetophenone phenyl hydrazine	Skin Contact eye contact	Flush immediately with plenty of H ₂ O
polyposphoric acid	Causes redness pain and poor visibility	Flush the eye and skin with water
2-phenyl indole	Causes eyes and skin irritation Hemifall if swallowed	Flush the eyes and skin with water for 15 mint

FOR EDUCATIONAL USE

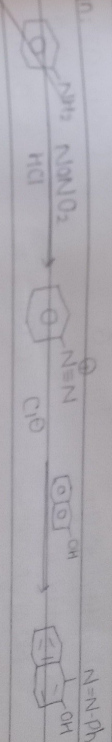
2-Naphthol \rightarrow 1-Phenyl-azo-2-naphthol \rightarrow 1-Amino-2-naphthol

part-I

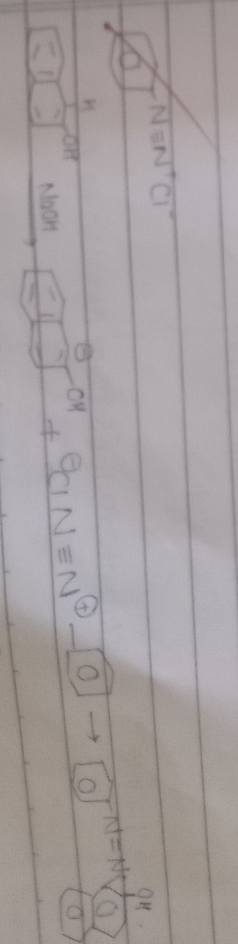
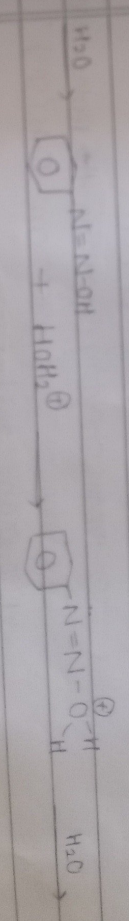
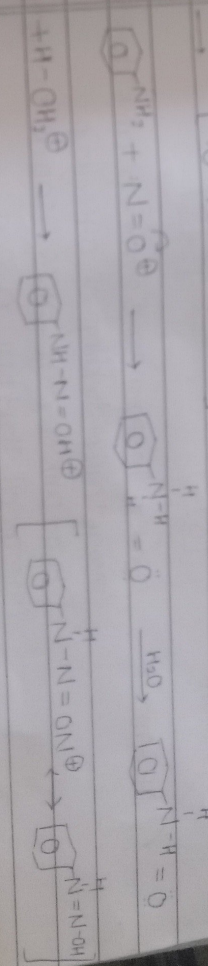
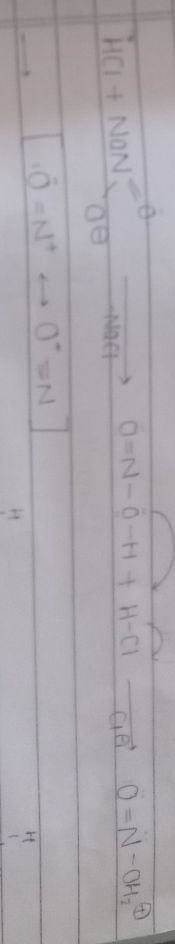
Aim: preparation of 1-phenyl-azo-2-naphthol from 2-naphthol

Requirements: Conical flask, 2-naphthol, conc. HCl, NaOH solⁿ etc

Reaction:



Mechanism:



FOR EDUCATIONAL USE

RF value = $\frac{\text{Distance traveled by solute}}{\text{Distance traveled by solvent}}$

$$= \frac{3.4}{4.8}$$

$$= 0.74$$

Weight of the product: 0.49 gm

Theoretical yield: 0.919 gms

practical yield: 43.53%

physical Constant: 190°C

RF value: non-polar 80%
polar 20%

MSDS Data:

Compound.	Health Hazards	First Aid
Acetophenone	Skin Contact	Flush immediately
phenyl hydrazine	eye Contact	With plenty of water
polyphosphoric acid	Causes redness pain and poor visibility	Flush the eye and skin with water
2-phenyl indole.	Causes eyes and skin irritation Harmful if swallowed	Flush the eyes and skin in with water for 15 mint

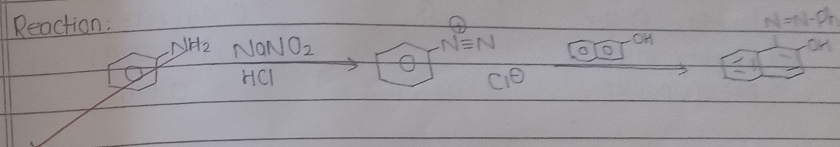
2-Naphthol \rightarrow 1-phenyl-azo-2-naphthol \rightarrow 1-Amino-2-naphthol

part-I

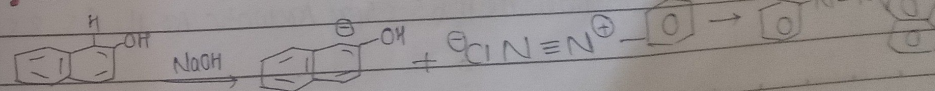
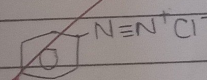
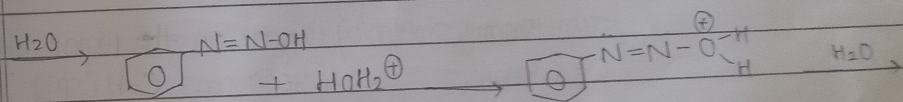
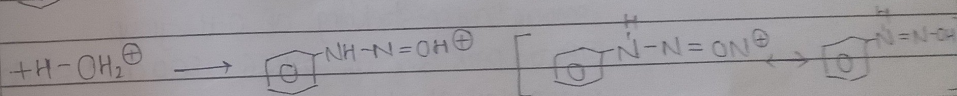
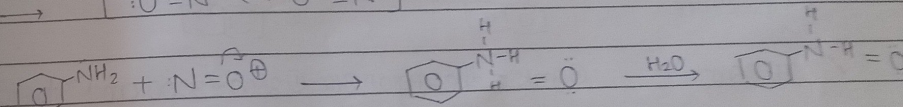
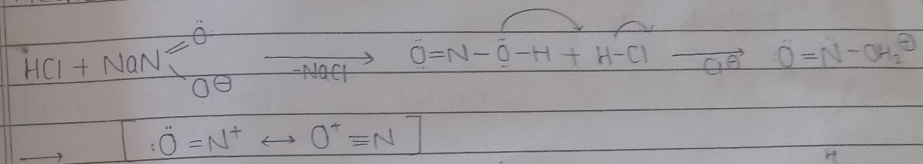
Aim: preparation of 1-phenyl azo-2-naphthol From 2-naphthol

Requirements: Conical flask, 2-naphthol, aniline, NaOH solⁿ etc.

Reaction:



Mechanism:



FOR EDUCATIONAL USE

Stoichiometric Calculation:

	2-Naphthol	Aniline (1.0297 g/cm ³)
Molecular formula	C ₁₀ H ₈ O	C ₆ H ₇ N
Molecular weight	144	93
Molar ratio	1	1
Quantity in gm	6	0.041 x 93 = 3.81
Quantity in cm ³	-	3.81 - 1.0297 = 3.7
No of Moles	0.041	0.041

Spectral Data:

	2-naphthol.	1-phenyl-2-azo-2-naphthol
Structure		
IR cm ⁻¹	<p>① peak in the region 3100-3000 cm⁻¹; C-H sp² stretching of O-H group.</p> <p>② Broad peak 3600 cm⁻¹ 3200 cm⁻¹ OH stretching of C=C.</p> <p>③ 1650-1500 cm⁻¹ stretching C=C atom.</p> <p>④ 1300-1050 cm⁻¹ stretching C=O group.</p>	<p>① 3600-3200 cm⁻¹ stretching of O-H group.</p> <p>② 1666-1500 cm⁻¹ stretching of C=C.</p> <p>③ peak in the region 3100-3000 cm⁻¹ C-H (sp²)</p>

¹H NMR

Aromatic Ar-H: 6.5-8.5 ppm Aromatic Ar-H: 6.5-8.5 ppm

procedure: Dissolve 4 cm³ of aniline in 25 cm³ of 11 HCl. Diazotise this mixture by addition of solution of sodium nitrite (3.5 g of sodium nitrite in 14 cm³ of water). Maintain the temperature of the reaction mixture below 5°C

prepare a solution of 6g of 2-naphthol in 35 cm³ of 10% NaOH solⁿ and cool this solution to 5°C stir the Naphthol solution vigorously and to it, add cold diazonium salt solution has been added, allow the mixture to stand in ice bath for 30 min. with occasional stirring. filter the product using Buchner funnel, wash the residue with water and dry. Note the yield of Crude 1-phenyl 2-azo-2-naphthol. purify small portion and take melting point.

Observation:

Weight of Crude product: 8.00g

Melting point of the product: 135°C

Calculation:

Theoretical yield:

93 g of aniline = 248 g of 1-phenyl 2-azo-2-naphthol

3.81 g of aniline = x g of 1-phenyl 2-azo-2-naphthol

$$93 \times x = 248 \times 3.81$$

$$x = \frac{248 \times 3.81}{93}$$

$$x = 10.16 \text{ g.}$$

percentage yield:

$$\frac{\text{observed yield}}{\text{Theoretical yield}} \times 100$$

$$\frac{8.00}{10.16} \times 100$$

78.74%

Result:

Weight of product (Theoretical yield): 8.00 g.

Theoretical yield: 10.16 g

percentage yield: 78.74%

physical constant: 135°C

MSDS Data:

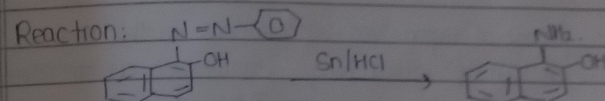
Name of Compound	Health Hazards	First aid
1-Aniline	It causes skin irritation. Synthetic effects occur from all routes of exposure and can include.	Quickly remove contaminated clothing. Wash contaminated skin with large amounts of soap and water.
	Causes eye, skin irritation may cause irritation of the digestive tract respiratory tract irritation.	Immediately flush skin with plenty of water for at least 15 minutes. Get medical aid immediately.

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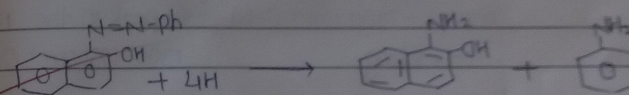
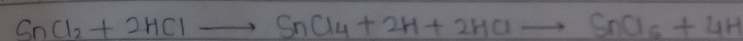
part-II

Aim: preparation of 1-amino-2-naphthol from 1-phenyl azo-2-naphthol

Requirements: RBF, chloride, alcohol, 1-phenyl azo-2-naphthol, etc.



Mechanism:



Stoichiometric calculation:

	1-phenyl-azo-2-naphthol	Tin (II) chloride
Molecular formula	$\text{C}_{16}\text{H}_{13}\text{NO}$	SnCl_2
Molecular weight	248	189
Molar ratio	1	1
Quantity in gm	1	$0.004 \times 189 = 0.75$
No. of Moles	0.004	0.004

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Spectral Data:

	1-phenyl azo-2-naphthol	1-amino-2-naphthol
Structure		
IR cm^{-1}	<p>@peak at region 3100-3000 cm^{-1} C-H sp^2 stretching</p> <p>@Broad peak 3600-3200 cm^{-1} OH stretching.</p> <p>@peak in the region 1680-1500 cm^{-1} C=C stretching</p> <p>@peak $\sim 1200 \text{ cm}^{-1}$ C-N stretching.</p> <p>6.5-8.5 ppm Aromatic ring present.</p> <p>4.8 ppm Aromatic OH proton.</p>	<p>@peak in the region 3100-3000 cm^{-1} C-H sp^2 stretching</p> <p>@Broad peak 3600 cm^{-1} N-H stretching.</p> <p>@peak in the region 1680-1500 cm^{-1} C=C stretching</p> <p>@peak in the region 1300-1050 cm^{-1} C=O stretching.</p> <p>6.5-8.5 ppm Aromatic ring present.</p>

procedure: Reflux the mixture of 1gm of 1-phenyl azo-2-naphthol and 10 cm^3 of rectified Spirit till the azo compound is dissolved. Separately prepare a reducing mixture of 6.7 g $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ in 20 cm^3 Conc HCl. add this reducing mixture to 1-phenyl azo-2-naphthol and reflux for further 30 mint. the solution turns a brown colour. Decant the solution into a beaker and place in ice bath till 1-amino-2-naphthol hydrochloride separates as greyish white white crystals. filter the product Using suction.

Wash with dil HCl and dry. Note the yield of 1-amino-2-naphthol and take the M.P of product and check TLC.

Observation:

Weight of crude product: 0.4g
Melting point of the product: 250°C

Calculation:

Theoretical yield:
248 g of 1-phenyl azo-2-naphthol = 15g of 1-amino-2-naphthol
1g of 1-phenyl azo-2-naphthol = x g of 1-amino-2-naphthol
$$\frac{248 \times x}{15} = 159 \times 1$$
$$x = \frac{159 \times 1}{248}$$

$$x = 0.64 \text{ g.}$$

percentage yield:

$$= \frac{\text{observed yield}}{\text{Theoretical yield}} \times 100$$

$$= \frac{0.4}{0.6} \times 100$$

$$= 62.5\%$$

Rf Value: $\frac{\text{Distance traveled by Solute}}{\text{Distance traveled by solvent}}$

$$= \frac{3.2}{3.5} = 0.9$$

Result:

weight of the product (observed yield) = 0.4g

Theoretical yield = 0.6g

percentage yield = 62.5%

physical constant (M.P) = 250°C

RF value = 0.9

(solvent system = 90% n-hexane and 10% ethyl acetate)

MSDS Data:

Name of Compound	Health Hazards	First aid.
① Tin (II) chloride	① Cause eye burns ② Cause skin burns ③ Harmful if swallowed	Flush the eye and skin with plenty of water. Get medical aid immediately.
HCl	very hazardous in cause of ignition	flush the eye and skin with cold water.
Alcohol	Causes eyes, skin and respiratory tract irritation	flush eye and skin with plenty of water.
2014127 1-phenyl-2-azo-2-naphthol	may cause an allergic skin reaction suspected of causing cancer	flush the skin with plenty of water get medical immediately

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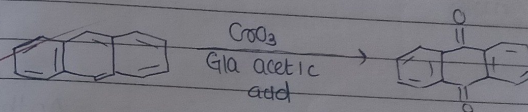
Anthracene → Anthraquinone → Anthrone.

part-I

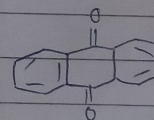
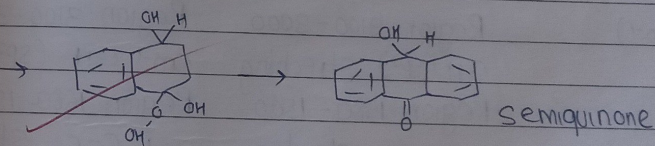
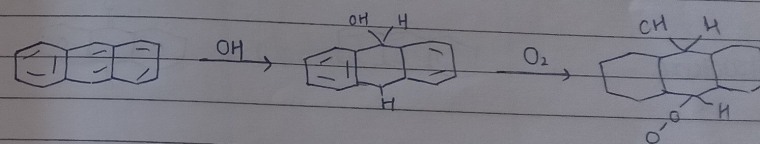
Aim: preparation of anthraquinone from Anthracene

Requirement: Beakers, glacial acetic acid, CrO_3 , Anthracene, Cold water

Reaction:



Mechanism:



① quinone (9,10) anthra

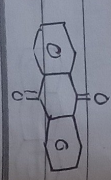
FOR EDUCATIONAL USE

- 100 g benzoin + 200 cm³ conc HNO₃ in capitol
- heat on boiling H₂O bath until 300 cm³ cold
- 26/04/24

Spectrometric Calculation:-

	Anthracene	Anthraquinone
Molecular formula	C ₁₄ H ₁₀	C ₁₄ H ₈ O ₂
Molecular weight	178	210
Molar Ratio	55	2.89
Q (gm)	-	-
Q (cm ³)	0.028	0.028
no of moles		

Structure



IR (cm⁻¹)

Anthracene	Anthraquinone
Region 3100-3000 C-H (sp ²) stretching	Region 3100-3000 C-H (sp ²) stretching
Region 1680-1500 C=C stretching	Region 1680-1500 C=C stretching

¹H NMR

Anthracene	Anthraquinone
Aromatic C-H at region 6.5-8.5 ppm	Aromatic C-H at region 6.5-8.5 ppm

procedure:-

Dissolve 5 grams of anthracene by refluxing it with 50 cm³ of glacial acetic acid.
prepare solution of 2.2 gms of CrO₃ in 25 cm³ of glacial acetic acid.

Add the oxidising agent to the anthracene soln and then reflux for further 8-10 minutes.
When all anthracene get oxidised completely cool the reaction.

Mixture and pour into 250 cm³ of cold water with stirring
The product is separate by filtration wash with hot water and dry

Note the yield of crude Anthraquinone purify the small portion of the product and take it mp.

Observation:-

Weight of the product: 4.7 gms
melting point : 283°C

Calculations:-

Theoretical yield:-

78 gm of Anthracene → 208 gm of Anthraquinone.
3 gm of Anthracene → x gm of Anthraquinone

$$x = \frac{208 \times 3}{78}$$

$$x = 5.84 \text{ gm.}$$

26/04/24
 24 benzoin - 2.2g conc HCl in conical
 1. adding H₂O bath 50 ml

percentage yield:- $\frac{\text{weight of the product}}{\text{theoretical yield}} \times 100$

$$= \frac{4.7}{5.84} \times 100 = 80\%$$

Result:-

Theoretical yield:- 5.84 gm
 weight of product:- 4.7
 Percentage yield:- 80%
 physical constant: 283°C.

Name of Compound.	Health Hazards.	First Aid.
Anthraquinone	eye contact skin contact inhalation.	Rinses immediately with plenty of Rins with plenty of water at least 15 minutes get attention move to the fresh air
Glacial acetic acid	eye contact skin contact inhalation.	Rinse cautiously with water for several min. Remove lenses take of immediately all contaminated clothing Rinse skin with water.

Anthraquinone

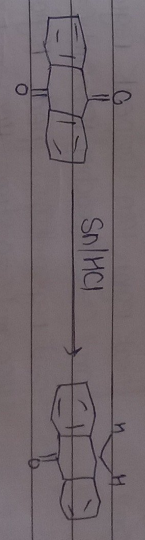
Causes eyes and skin irritation. may also causes respiratory irritation	Immediately flush skin with plenty of water get medical attention. Immediately flush eyes with plenty of water more to fresh air if not breathing give artificial respiration.
Inhalation	

Part-II

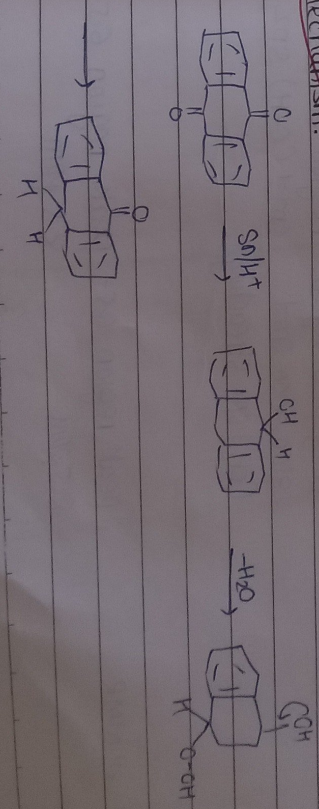
Aim preparation of Anthrone from Anthraquinone.

Requirements: Beakers, glacial acetic acid, Anthraquinone, conc HCl

Reaction:



Mechanism:



after removal from benzoin
- 4g benzoin + 30 cm³ conc HNO₃ in conical
- heat in boiling H₂O bath until acid is
- cold in cold water

Stoichiometric calculation:	
Anthraquinone	Tin
Molecular formula	C ₁₄ H ₈ O ₂
Molecular weight	208
Molar ratio	1
Q (gm)	1 gm
no. of moles	0.0048

Spectral Data:

Anthraquinone Anthracene



IR cm⁻¹

peak region 1680-1500 cm ⁻¹ C=C stretching	peak region 1680 cm ⁻¹ C=C stretching
peak region 1800-1650 cm ⁻¹ C=O stretching	peak region 1800-1650 cm ⁻¹ C=O stretching
OH peak region 3100-3000 cm ⁻¹ (CH) sp ² stretching	region 3100-3000 cm ⁻¹ (C-H) sp ²

¹H NMR

peak region 6.5-8.5 ppm	peak region 6.5-8.5 ppm
-------------------------	-------------------------

Procedure:-

Reflux the reaction mixture of 1 gm anthraquinone 5 grams of granulated tin and 35 cm³ of glacial acetic acid for 15-20 min
cool and then add dropwise 13 cm³ of conc HCl to it.
If all anthraquinone does not dissolve than add some more granulated tin and HCl
Filter and wash with water and dry by pressing between the filter paper.
Recrystallize it from a mixture of benzene and petroleum ether
Note the yield of pure Anthracene. take the TLC and dride product.

Observation:-

Weight of product:- 0.2 gm
physical Constant :- 155°C

Calculation

Theoretical yield:-

208 gms of Anthraquinone = 144 gms of Anthracene
1 gms of Anthraquinone = x gms of Anthracene

$$x = \frac{144 \times 1}{208}$$

$$x = 0.933 \text{ gm.}$$

$\text{wt. of } \text{H}_2\text{SO}_4 = 2.2 \text{ gms}$ conc H_2SO_4 is added
 - heat on boiling H_2O bath for 1 hr
 - 20/04/14

percentage yield :- $\frac{\text{weight of product}}{\text{theoretical yield}} \times 100$

$$= \frac{0.2}{0.933} \times 100$$

$$= 21.4\%$$

RF value: Distance travelled by solute
 Distance travelled by solvent

$$= \frac{5.3}{6.2}$$

$$= 0.85$$

Result:

weight of the product :- 0.2 gms

Theoretical yield :- 0.933 gms

percentage yield :- 21.4%

RF value :- 0.85

physical constant :- 155°C

solvent system :- n-hexane = 60

ethyl acetate = 40.

MSDS Data:-

Name of Compound.	Health Hazard	First Aid
Anthraquinone.	Skin Contact eyes contact Inhalation	Immediately flush skin with plenty of immediately flush with plenty of for at least 15 min
glacial acetic acid.	Skin contact eyes contact Inhalation.	Rinses with water for several min remove contact lenses. Immediately flush eyes.

h
 26/4/15

- 100 mg benzoin + 20 ml conc HCl in conical flask
 - heat on boiling H₂O bath for 30 min
 - 26/04/24

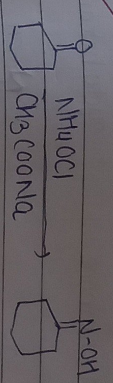
Pract. exp. No. 6,
 Part - I

cyclohexanone \rightarrow cyclohexanone oxime \rightarrow caproactum.

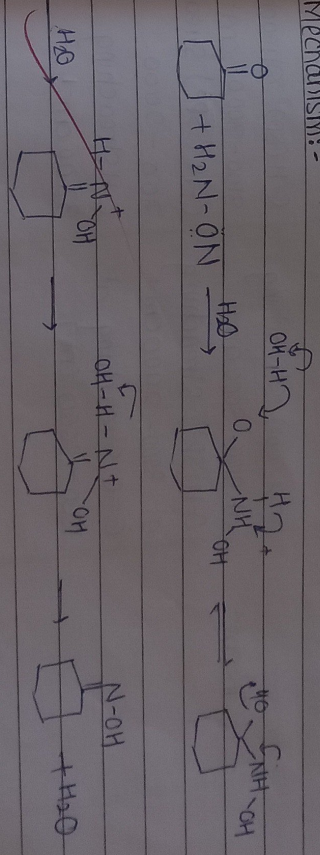
Aim:- preparation of cyclohexanone oxime from cyclohexane.

Requirements:- Beaker, ice bath, hydroxylamine,
 hydrochloride sodium acetate, distilled water
 cyclohexane

Reaction:-



Mechanism:-



Practical - 2 Preparation of benzil from benzoin

26/04/24

- 4g benzoin + 20cm³ conc HNO₃ in conical
- heat on boiling H₂O bath until evap.
- 20cm³ cold H₂O

Stoichiometric Calculation:-

	cyclohexanone	NH ₄ OCl
Molecular formula	C ₆ H ₁₀ O	NH ₄ OCl
Molar ratio	1	3.312
W (gm)	4.7 gms	-
W (cm ³)	5 cm ³	0.048
no. of moles	0.048	

Spectral Data:

	cyclohexanone	cyclohexanone oxime
Structure		
IR cm ⁻¹	<p>C-H (sp²) Streaching at 3100-3000 cm⁻¹</p> <p>C-H (sp³) Streaching at 3000-2900 cm⁻¹</p> <p>C=O Streaching at 1800-1660 cm⁻¹</p>	<p>C-H (sp²) Streaching 3100-3000 cm⁻¹</p> <p>C-H (sp³) Streaching 3000-2900.</p> <p>C-H Streaching 3200 cm⁻¹</p>
H ¹ NMR	Ar-H at region 6.5-8.5 ppm	Ar-H at region 6.5-8.5 ppm

FOR EDUCATIONAL USE

procedure:-

Dissolve mixture of 3.5 gms of hydroxylamine hydrochloride and 4.2 gms of sodium acetate in 20cm³ of water to this solution add 5 cm³ of cyclohexanone in small portion. Shake the flask well and cool it in cold water. Crystals of cyclohexanone oxime are formed. Collect the crystals by filtering cold solution using Buchner Funnel.

Wash the crystals with small portion of cold water then dry in air.

Note the yield of crude cyclohexanone oxime and purify the small portion of product and take melting point.

Calculation:-

Theoretical yield:-

98 gms of cyclohexanone = 113 gm of cyclohexanone oxime
4.74 gms of cyclohexanone = x gm of cyclohexanone oxime

$$x = \frac{113 \times 4.74}{98}$$

$$x = 4.7 \text{ gms.}$$

Percentage yield = $\frac{\text{yield of the product}}{\text{Theoretical yield}} \times 100$

$$= \frac{3.2}{5.47} \times 100$$

$$= 64\%$$

FOR EDUCATIONAL USE

Result:-

Theoretical yields:- 5.47gms

Weight of the products:- 3.2gms

Percentage yield:- 64%

melting point :- 88°C

MSDS Data:-

Compound	Health Hazards	First Aid
cyclohexanone	eyes contact skin contact inhalation	Rinse immediate with plenty of water Wash of immediately with plenty of water move to the fresh air and get medication.
Sodium acetate	Skin Contact eye Contact swallowing	Wash affected area with soap and water protect exposed eyes. Remove lenses. Rinse mouth thoroughly discomfort or vomiting persists.
Hydroxylamine hydrochloride	Skin contact eye contact ingestion	Wash off immediately with plenty of water Rinse immediately with plenty of water Do not induce vomiting.

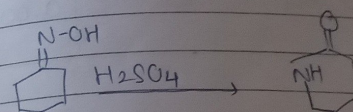
part-II

26

Aim: preparation of caprolactum from cyclohexanone oxime

Requirement: cold water, ice bath, beaker, H₂SO₄, cyclohexanone oxime, NaOH etc.

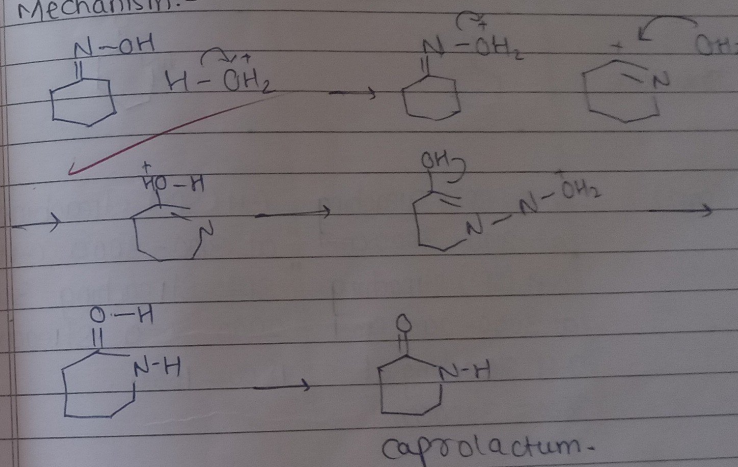
Reaction



cyclohexanone oxime

caprolactum.

Mechanism:-



Practical - 1 Preparation



Part - I: Preparation of benzil from benzoin

26/04/24

- 4g benzoin + 20 cm³ conc HNO₃ in conical
- heat on boiling H₂O bath until
- 10 cm³ cold

Stoichiometric	cyclohexanone.	conc H ₂ SO ₄
Molecular formula	C ₆ H ₁₀ NO	H ₂ SO ₄
Molecular Weight	113	98
Molar ratio	1	1
W (gm)	19m	0.8624
W (cm ³)	-	0.468
no of moles	0.0088	0.0088

Spectral Data

	cyclohexanone Oxime	caprolactum
Structure		
IR (cm ⁻¹)	C-H (sp ²) stretching at 3100-3000 cm ⁻¹ C-H (sp ³) stretching at 3000-2900 cm ⁻¹ O-H stretching at 3600-3200 cm ⁻¹	C-H (sp ²) stretching at 3100-3000 C-H sp ³ stretching 3000-2900 C=O stretching 1800-1660.
¹ H NMR	Aromatic H at region 6.5-8.5 ppm	Aromatic H at region 6.5-8.5 ppm.

procedure:-

Take 1g of cyclohexanone Oxime add cold solution of conc. H₂SO₄. Warm the reaction mixture gently to bring the reaction to begin the reaction.

Remove the flask from burner and allow the continue. Once the reaction is subsite cool the content of flask and then 10 cm³ of water.

mean while prepare a solution of 2.2 g of NaOH in 7.2 cm³ of water. add prepared solution to the reaction mixture dropwise transfer this cold alkaline solution to the separating funnel and extract the compound by using three portion of 5 cm³ of carbon tetrachloride yellow oil obtained which is solidified cooling the purified compound.

Note the yield of the pure caprolactum. take mp.

Check the purity of by TLC submit the crude product.

Observation:-

Weight of the purified product: 0.8 gm.

Melting point :- 66°C

Calculation

Theoretical yield

113 g of cyclohexanone oxime = 113 gm of caprolactum
 1 g of cyclohexanone oxime = x gm of caprolactum

$$x = \frac{113 \times 1}{113}$$

$$x = 1 \text{ gms.}$$

Practical - 2 Preparation
Part - I Preparation of benzil from benzoin

DATE: 26/04/24

- 4g benzoin + 20cm³ conc HNO₃ in conical flask
Heat on boiling H₂O bath until
20cm³ cold

Percentage yield: $\frac{\text{weight of the product}}{\text{Theoretical yield}} \times 100$

$$= \frac{0.8}{1} \times 100$$

$$= 80\%$$

Rf value: $\frac{\text{Distance travelled by solute}}{\text{Distance travelled by solvent}}$

$$= \frac{4.5}{3.2} = 1.4$$

Result:

Theoretical yield: 1 gms

Weight of product: 0.8 gms

Percentage yield: 80%

Melting point: 66°C

Rf Value: 1.4

Solvent system: ethyl acetate 20%
n-hexane: 80%

28

MSDS Data:

Compound	Health Hazards	First aid
Caproic Acid	Skin Contact Inhalation Ingestion	Wash immediately with soap and remove to fresh clean mouth with water.
Carbon tetrachloride	eye contact Inhalation Ingestion	Wash immediately plenty of water move to fresh air do not induce vomine
H ₂ SO ₄	eye contact Skin contact Inhalation.	Rinse immediately with plenty of water wash immediately and move to fresh air

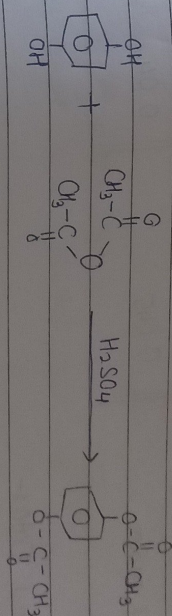
26/4/23

Hydroquinone \rightarrow Hydroquinone diacetate \rightarrow 2,5-dihydroxyacetophenone

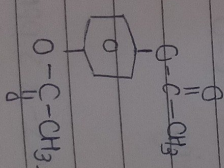
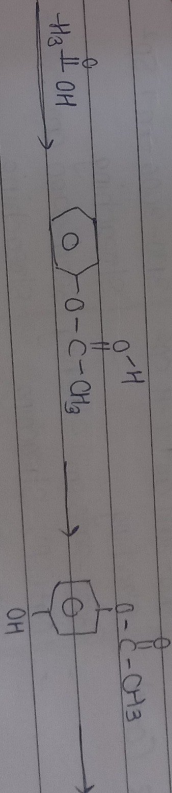
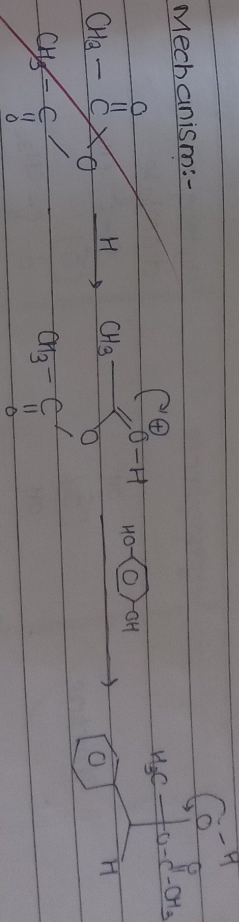
Aim:- Preparation of hydroquinone diacetate from hydroquinone.

Requirements:- Conical flask, glass rod, Conc. H_2SO_4 , hydroquinone, acetic anhydride, cold water.

Reaction:-



Mechanism:-



4g benzoin + 20cm³ conc HNO₃ in conical flask
 heat on boiling H₂O bath for 1 hr
 20cm³ cold water added

Stoichiometric Calculation:-

	hydroquinone	Acetic anhydride
Molecular formula	C ₆ H ₆ O ₂	C ₄ H ₆ O ₃
Molecular weight	110	102
molar ratio	1	1
Q (gm)	5	4.59
Volume	-	4.25
no of moles	0.045	0.04

Spectral Data:-

Structure	hydroquinone	hydroquinone diacetate
IR (cm ⁻¹)	3100-3000 cm ⁻¹ C-H sp ² Stretching 3000-2900 cm ⁻¹ Stretching C-H (sp ³) 3500-3200 cm ⁻¹ O-H group	3100-3000 cm ⁻¹ sp ² Stretching 3000-2900 cm ⁻¹ Stretching 1800-1650 C=O Stretching
¹ H NMR	Ar-H 6.5-8.5 ppm R-OH 3-6.5 ppm	Ar-H 6.5-8.5 ppm

Procedure:-

Add a 1 drop of conc. H₂SO₄ to a mixture of 5 gm of hydroquinone and 8-6 cm³ of acetic anhydride in conical flask.
 Stir the mixture until dissolve hydroquinone.
 After 5 minutes pour the mixture into crushed ice.
 Filter with suction pump and wash with cold water. Note the yield of hydroquinone diacetate.
 Purify the small portion of product and take melting point.

Observation:-

Weight of product:- 3.8 gm
 melting point 8-124°C

Theoretical yield:-

110 gm of hydroquinone = 102 gm of hydroquinone diacetate.
 5 gm of hydroquinone = x

$$x = \frac{102 \times 5}{110}$$

$$x = 4.62$$

percentage yield:-

Weight of the product x 100
 Theoretical yield

$$\frac{5.8}{8.82} \times 100$$

$$= 70\%$$

Result:-
 Weight of product:- 5.82 gm
 Theoretical yield:- 8.82 gm
 Percentage yield:- 70%
 Melting point:- 8-104°C

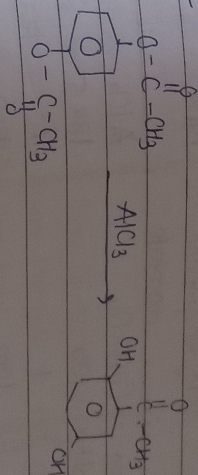
MSDS Data:-		
Name of compound	Health Hazard	First Aid.
Hydroquinone	Skin Contact Eye Contact Ingestion.	Wash immediately with plenty of water and remove contact lenses immediately. Flush eyes with water.
Azobenzene anhydride	Skin Contact Eye Contact Inhalation	Wash immediately with water. Take immediately medical treatment flush eyes with plenty of water.

Part-II

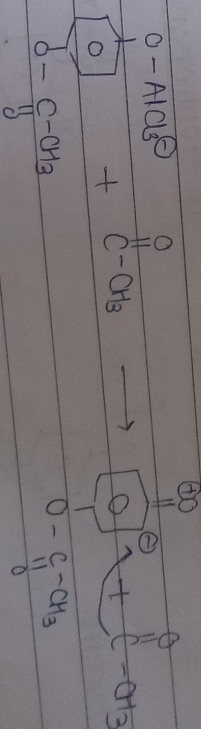
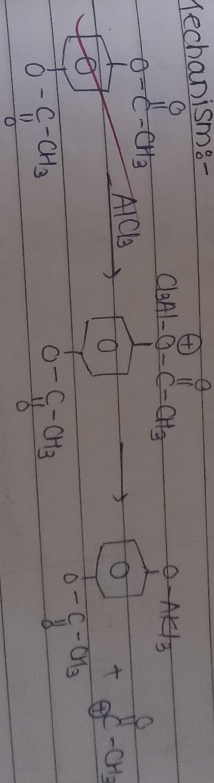
Aim:- preparation of 2,5-dihydroxy acetophenone from hydroquinone diacetate.

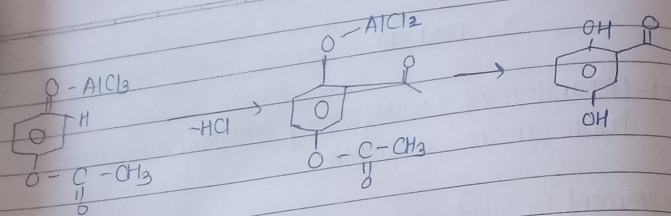
Requirement:- Glass rod, Conical flask, hydroquinone diacetate, NaOH , Conc. HCl .

Reaction:-



Mechanism:-





Stoichiometric calculation:-

	Hydroquinone diacetate	AlCl ₃
Molecular formula	C ₁₂ H ₁₀ O ₄	AlCl ₃
Molecular weight	194	133.5
Molar ratio	1	1
Q (gm)	1gm	0.629
Volume (cm ³)	-	-
no of moles.	0.0051	0.0051

Spectral Data:-

	Hydroquinone diacetate	2,5-dihydroxyacetophenone
Structure:		

IR (cm⁻¹)

3100-3000 cm ⁻¹ CH(sp ²)	3100-3000 cm ⁻¹ CH(sp ²)
3000-2900 cm ⁻¹ CH(sp ³)	3000-2900 cm ⁻¹ CH(sp ³)
1800-1660 cm ⁻¹ C=O	1660-1650 cm ⁻¹ C=O

¹H NMR

Ar-H 6.5-8.5 ppm	Ar-H 6.5-8.5 ppm.
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procedure:-

Take the mixture of 1gm of hydroquinone diacetate and 2.5 gm of anhydrous aluminium chloride in rbf and fitted by air condenser by calculation tube.

Heat the flask in oil bath slowly such that the temperature reaches 100-120 at the end of 30 min evolution of hydrogen chloride the begins

Now rise the temperature slowly to 160-165°C maintain the temperature for 3 hours.

allow to cool at room temperature.

Add crushed ice followed by 20° cm HCl in excess deluminate to excess of AlCl₃.

Filter the product with suction and wash it with cold water

Recrystallize with 99% ethanol

Note the yield of product.

check the purity by TLC and take melting point

Submit the dried product.

Observation:-

Weight of product:- 0.4

Melting point :- 204°C .

Calculation:-

Theoretical yield:-

194 gm of hydroquinone diacetate = 152 gm 2,5 dihydroxy acetophenone

1 gm of hydroquinone diacetate = $\frac{x}{152}$ gm 2,5 dihydroxy acetophenone.

$$x = \frac{194 \times x}{152}$$

$$x = \frac{194}{152}$$

$$x = 1.3 \text{ gms}$$

percentage yield:-

$$\frac{\text{Weight of the product}}{\text{Theoretical yield}} \times 100$$

$$= \frac{0.4}{1.3}$$

$$= 30.7\%$$

RF value:- $\frac{\text{Distance travelled by solute}}{\text{Distance travelled by solvent}}$

$$= \frac{3.5}{4.2}$$

$$= 0.83$$

Solvent system:- non-polar - 60%
polar - 40%

Result:-

Weight of the product:- 0.4

Theoretical yield :- 1.3 gms

percentage yield :- 30.7%

RF value :- 0.83.

Solvent system :- non-polar - 60%
polar - 40%

Melting point :- 204°C

Observation:-

Weight of products - 0.4
Melting point - 204°C .

Calculations:-

Theoretical yields:-

1.94 gm of hydroquinone diacetate = 152 gm 2,5 dihydroxy acetophenone

1 gm of hydroquinone diacetate = $\frac{x}{152}$ gm 2,5 dihydroxy acetophenone.

$$x = \frac{1.94 \times x}{152}$$

$$x = \frac{1.94}{152}$$

$$x = 1.3 \text{ gms}$$

percentage yield:-

$$\frac{\text{Weight of the product}}{\text{Theoretical yield}} \times 100$$

$$= \frac{0.4}{1.3}$$

$$= 30.7\%$$

RF value:- $\frac{\text{Distance travelled by solute}}{\text{Distance travelled by solvent}}$

$$= \frac{3.5}{4.2}$$

$$= 0.83$$

Solvent system:- non-polar - 60%
polar - 40%

Result:-

Weight of the products - 0.4

Theoretical yield :- 1.3 gms

percentage yield :- 30.7%

RF value :- 0.83

Solvent system :- non-polar - 60%
polar - 40%

Melting point :- 204°C

MSDS Data:-

Name of Compound	Health Hazard	First Aid
Hydroquinone diacetate	eyes contact skin contact	Wash with plenty of water and move to fresh air.
Aluminium Chloride	eyes Contact skin Contact	Wash with plenty of water and take medical treat ment and move to fresh air

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